

June 24, 2016

Tom Moe  
USS Corporation  
P.O. Box 417  
8771 Park Ridge Dr  
Mountain Iron, MN 55768

RE: Project: NPDES-TB WK 3  
Pace Project No.: 1268349

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on June 15, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,



Melisa M Woods  
melisa.woods@pacelabs.com  
Project Manager

Enclosures

cc: Cory Hertling  
Terri Sabetti, NTS



## REPORT OF LABORATORY ANALYSIS

This report shall not be reproduced, except in full,  
without the written consent of Pace Analytical Services, Inc..

## CERTIFICATIONS

Project: NPDES-TB WK 3

Pace Project No.: 1268349

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### Virginia Minnesota Certification ID's

315 Chestnut Street, Virginia, MN 55792

Alaska Certification #MN01084

Arizona Department of Health Certification #AZ0785

Minnesota Dept of Health Certification #: 027-137-445

North Dakota Certification: # R-203

Wisconsin DNR Certification # : 998027470

WA Department of Ecology Lab ID# C1007

Nevada DNR #MN010842015-1

Oklahoma Department of Environmental Quality

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### Duluth Minnesota Certification ID's

4730 Oneota St., Duluth, MN 55807

Minnesota Dept of Health Certification #: 027-137-152

Wisconsin DNR Certification # : 999446800

North Dakota Certification #: R-105

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## REPORT OF LABORATORY ANALYSIS

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## SAMPLE SUMMARY

Project: NPDES-TB WK 3

Pace Project No.: 1268349

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1268349001	SD 001 (Seep 020)	Water	06/15/16 11:15	06/15/16 14:30

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## SAMPLE ANALYTE COUNT

Project: NPDES-TB WK 3

Pace Project No.: 1268349

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1268349001	SD 001 (Seep 020)	EPA 1664 TPH	BT1	1	PASI-DUL
		USGS I-3765	BEM	1	PASI-V

## REPORT OF LABORATORY ANALYSIS

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## ANALYTICAL RESULTS

Project: NPDES-TB WK 3

Pace Project No.: 1268349

Sample: SD 001 (Seep 020)		Lab ID: 1268349001		Collected: 06/15/16 11:15		Received: 06/15/16 14:30		Matrix: Water	
Parameters	Results	Units	Report Limit	MDL	DF	Prepared	Analyzed	CAS No.	Qual
<b>1664 SGT-HEM, TPH</b>									
		Analytical Method: EPA 1664 TPH							
Total Petroleum Hydrocarbons	ND	mg/L	3.0	0.91	1		06/20/16 17:58		
<b>USGS I-3765 TSS</b>									
		Analytical Method: USGS I-3765							
Total Suspended Solids	3.6	mg/L	1.0	1.0	1		06/21/16 12:58		

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: NPDES-TB WK 3

Pace Project No.: 1268349

QC Batch: DUL/7601

Analysis Method: EPA 1664 TPH

QC Batch Method: EPA 1664 TPH

Analysis Description: 1664 SGT-HEM, TPH

Associated Lab Samples: 1268349001

METHOD BLANK: 332203

Matrix: Water

Associated Lab Samples: 1268349001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Petroleum Hydrocarbons	mg/L	ND	3.0	0.90	06/20/16 16:40	

LABORATORY CONTROL SAMPLE: 332204

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	20	15.0	75	64-132	

MATRIX SPIKE SAMPLE: 332205

Parameter	Units	1268463001 Result	Spike Conc.	MS Result	MS % Rec	% Rec Limits	Qualifiers
Total Petroleum Hydrocarbons	mg/L	1.4J	21.1	18.7	83	64-132	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA

Project: NPDES-TB WK 3

Pace Project No.: 1268349

QC Batch: WET/24753

Analysis Method: USGS I-3765

QC Batch Method: USGS I-3765

Analysis Description: USGS I-3765 Total Suspended Solids

Associated Lab Samples: 1268349001

METHOD BLANK: 332877

Matrix: Water

Associated Lab Samples: 1268349001

Parameter	Units	Blank Result	Reporting Limit	MDL	Analyzed	Qualifiers
Total Suspended Solids	mg/L	ND	1.0	1.0	06/21/16 12:57	

LABORATORY CONTROL SAMPLE: 332878

Parameter	Units	Spike Conc.	LCS Result	LCS % Rec	% Rec Limits	Qualifiers
Total Suspended Solids	mg/L	239	238	100	80-120	

SAMPLE DUPLICATE: 332879

Parameter	Units	1268501003 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	232	228	2	10	

SAMPLE DUPLICATE: 332880

Parameter	Units	1268425002 Result	Dup Result	RPD	Max RPD	Qualifiers
Total Suspended Solids	mg/L	28.0	26.0	7	10	

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.

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## QUALIFIERS

Project: NPDES-TB WK 3

Pace Project No.: 1268349

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### DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

### LABORATORIES

PASI-DUL Pace Analytical Services - Duluth

PASI-V Pace Analytical Services - Virginia

## REPORT OF LABORATORY ANALYSIS

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## QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: NPDES-TB WK 3

Pace Project No.: 1268349

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1268349001	SD 001 (Seep 020)	EPA 1664 TPH	DUL/7601		
1268349001	SD 001 (Seep 020)	USGS I-3765	WET/24753		

## REPORT OF LABORATORY ANALYSIS

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## Section A

**Required Client Information:**

Company:	USS Corporation
Address:	P.O. Box 417

Mt. Iron, MN 55768

Email:

Phone

Requested Due Date:

## Section B

**Required Project Information:**

Report To:	Tom Moe
Copy To:	

0	
1	
2	
3	
4	
5	
6	
7	
8	
9	

Purchase Order #:

Project Name: NPDES-TB Wk3

Project #:

## Section C

**Invoice Information:**

Attention:
Company Name

Address:

**Page Quote:**

Pace Project Manager: [heather.zika@pacelabs.com](mailto:heather.zika@pacelabs.com)

Page Profile #:

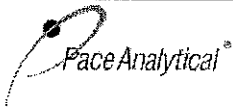
PM: MMH

**CLIENT: USS CORP**

**Due Date: 06/29/16**

1.

[illegible]

	Document Name:	Document Revised: 23Feb2015
	Sample Condition Upon Receipt Form	Page 1 of 1
	Document No.: F-VM-C-001-Rev.09	Issuing Authority: Pace Virginia, Minnesota Quality Office

**Sample Condition Upon Receipt**

Client Name:

*USG*

Project #:

**WO#: 1268349**



Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☒ Client  
☐ Commercial ☐ Pace ☐ Other: \_\_\_\_\_

Tracking Number: \_\_\_\_\_

Optional: Proj. Due Date: \_\_\_\_\_ Proj. Name: \_\_\_\_\_

Custody Seal on Cooler/Box Present? ☐ Yes ☒ No

Seals Intact? ☐ Yes ☒ No

Packing Material: ☐ Bubble Wrap ☐ Bubble Bags ☒ None ☐ Other: \_\_\_\_\_

Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ 140792808

Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 5.0

Cooler Temp Corrected °C: 5.3

Biological Tissue Frozen? ☐ Yes ☐ No ☒ N/A

Temp should be above freezing to 6°C

Correction Factor: 0.3

Date and Initials of Person Examining Contents: 6-15-16 *CL*

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
-Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: <i>WT</i>		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased):		

**CLIENT NOTIFICATION/RESOLUTION**

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

FECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review:

*M. Lisa Woods*

Date:

6/17/16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)

# Intra-Regional Chain of Custody

Workorder: 1268349

Workorder Name: NPDES-TB WK 3




Owner Received Date: 6/15/2016

Due Date: 6/29/2016

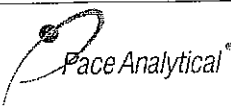
Pace Analytical Virginia  
315 Chestnut Street  
Virginia, MN 55792  
Phone (218) 742-1042

Pace Analytical Duluth  
4730 Oneota Street  
Duluth, MN 55807  
Phone (218) 727-6380

Report To:  
Melissa M Woods

Received at:		Send To Lab:		Preserved Containers		Requested Analysis		Comments																																				
Item	Sample ID	Sample Type	Collect Date/Time	Lab ID	Matrix	HCL																																						
1	SD 001 (Seep 020)	PS	6/15/2016 11:15	1268349001	Water	2																																						
2																																												
3																																												
4																																												
5																																												
<table border="1"> <thead> <tr> <th>Transfers</th> <th>Released By</th> <th>Date/Time</th> <th>Received By</th> <th>Date/Time</th> <th>Received on Ice</th> <th>Samples Intact</th> </tr> </thead> <tbody> <tr> <td>1</td> <td></td> <td>6/16/16</td> <td>Melissa M Woods</td> <td>6/16/16 15:45</td> <td></td> <td></td> </tr> <tr> <td>2</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> <tr> <td>3</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> <tr> <td>4</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> </tbody> </table>										Transfers	Released By	Date/Time	Received By	Date/Time	Received on Ice	Samples Intact	1		6/16/16	Melissa M Woods	6/16/16 15:45			2							3							4						
Transfers	Released By	Date/Time	Received By	Date/Time	Received on Ice	Samples Intact																																						
1		6/16/16	Melissa M Woods	6/16/16 15:45																																								
2																																												
3																																												
4																																												

\*\*\*In order to maintain client confidentiality, location/name of the sampling site, sampler's name and signature may not be provided on this COC document.  
This chain of custody is considered complete as is since this information is available in the owner laboratory.

	Document Name: <b>Sample Condition Upon Receipt Form</b>	Document Revised: 22Jan2016 Page 1 of 1
	Document No.: <b>F-DUL-C-001-Rev.01</b>	Issuing Authority: Pace Virginia, Minnesota Quality Office

**Sample Condition  
Upon Receipt**

Client Name:

Project #:

IR-COC

Courier: ☐ Fed Ex ☐ UPS ☐ USPS ☐ Client  
☐ Commercial ☒ Pace ☐ Other: \_\_\_\_\_

Tracking Number: \_\_\_\_\_

Custody Seal on Cooler/Box Present? ☒ Yes ☐ No

Seals Intact? ☒ Yes ☐ No

Optional: Proj. Due Date: \_\_\_\_\_ Proj. Name: \_\_\_\_\_

Packing Material: ☐ Bubble Wrap ☒ Bubble Bags ☐ None ☐ Other: \_\_\_\_\_

Temp Blank? ☒ Yes ☐ No

Thermometer Used: ☒ B00051

Type of Ice: ☒ Wet ☐ Blue ☐ None ☒ Samples on ice, cooling process has begun

Cooler Temp Read °C: 0.5

Cooler Temp Corrected °C: 1.1

Biological Tissue Frozen? ☐ Yes ☐ No ☒ NA

Temp should be above freezing to 6°C

Correction Factor: +0.6 °C

Date and Initials of Person Examining Contents: 6/16/16 Kp

Comments:

Chain of Custody Present?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	1.
Chain of Custody Filled Out?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	2.
Chain of Custody Relinquished?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	3.
Sampler Name and Signature on COC?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input checked="" type="checkbox"/> N/A	4.
Samples Arrived within Hold Time?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	5.
Short Hold Time Analysis (<72 hr)?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	6.
Rush Turn Around Time Requested?	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No <input type="checkbox"/> N/A	7.
Sufficient Volume?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	8.
Correct Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	9.
Pace Containers Used?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Containers Intact?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	10.
Filtered Volume Received for Dissolved Tests?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	12.
-Includes Date/Time/ID/Analysis Matrix: <u>WT</u>		
All containers needing acid/base preservation will be checked and documented in the pH logbook.	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	13.
Headspace in VOA Vials (>6mm)?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	14.
Trip Blank Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	15.
Trip Blank Custody Seals Present?	<input type="checkbox"/> Yes <input type="checkbox"/> No <input type="checkbox"/> N/A	
Pace Trip Blank Lot # (if purchased):		

**CLIENT NOTIFICATION/RESOLUTION**

Field Data Required? ☐ Yes ☐ No

Person Contacted: \_\_\_\_\_ Date/Time: \_\_\_\_\_

Comments/Resolution: \_\_\_\_\_

FECAL WAIVER ON FILE Y N

TEMPERATURE WAIVER ON FILE Y N

Project Manager Review:

AP for LMF

Date: 6-17-16

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e. out of hold, incorrect preservative, out of temp, incorrect containers)